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Evaluation of fire-retardant properties of edge-jointed lumber from tropical fast-growing woods using cone calorimetry and a standard fire test

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Abstract Some tropical fast-growing woods were converted to edge-jointed lumber, and their fire-retardant properties due to chemical coating were evaluated using cone calorimetry and a standard fire test. The woods used were Indonesian and Malaysian albizia and gmelina plantation trees, with Japanese hinoki as a reference. The lumber was coated with 100 g/m² of trimethylol melamine phosphoric acid in a 25% aqueous solution. The treated and untreated lumber was tested in a laboratory-scale exposure furnace in accordance with JIS A 1304 and the cone calorimeter test with heat flux of 40 kW/m² following the ISO 5660. Results showed that fire endurance of all lumber was enhanced by the treatment. The fire-retardant properties were improved with increasing surface density. Though a similar trend was seen, the fire-retardant properties of the lumber revealed by the cone calorimeter test were inferior to those seen with standard fire test. Addition of thermocouples to the cone calorimeter allowed us to obtain information on the critical temperature (260°C) and charring temperature (300°C) of the lumber.

Key words Tropical fast-growing woods \cdot Edge-jointed lumber \cdot Fire-retardant properties \cdot Cone calorimeter \cdot Standard fire test

Introduction

Albizia (*Paraserianthes falcataria* (L.) Nielsen) and gmelina (*Gmelina arborea* Roxb.) are fast-growing trees extensively planted in Indonesia and Malaysia. ¹ The woods show prom-

ise as new high-quality products with certain technological approaches.² Among the properties of the wood products, fire retardancy is important for fire safety. The combustibility of wood could be decreased by fire-retardant treatment. Many studies have been undertaken to improve the fire-retardant properties of wood and wood-based products (e.g., treatment using nitrogen compounds and phosphoric acid).³⁻¹⁰ Surface treatment using fire-retardant chemicals was shown to be a simple, effective, economical method.^{4,5,10}

Fire tests for wood-based products are mainly concerned with the surface properties of the products because their early fire behavior is important to many aspects of fire safety. Many standard fire tests have been used to evaluate the properties. Nowadays it is a world wide trend to use ISO standards. The cone calorimeter test (ISO 5660) has been widely accepted as a small-scale test and is considered the most useful and universal instrument, although standard fire tests such as the Japan Industrial Standard (JIS) A 1304, which is analogous to ISO 834, have also been widely used. However, the relationship of the JIS standard fire test to the ISO cone calorimeter test has not been clarified regarding practical application to the fire-retardant performance of wood-based products.

The purpose of this study was to evaluate the fire-retardant performance of edge-jointed lumber made from tropical fast-growing woods as determined by a standard fire test and the cone calorimeter test. To analyze the surface properties of the lumber, surface density (i.e., mass per unit area, or a product of density and thickness) is used. ^{14,15} This indicator is particularly effective, as it takes into account the effect of different densities and lumber thicknesses, as was observed in this study.

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Materials and methods

Materials and treatment

Wood blocks with dimensions of 300 (L) \times 10 (T) \times 10 (R) mm, 300 (L) \times 10 (T) \times 15 (R) mm, and 300 (L) \times 10 (T)

× 20 (R) mm, were prepared from albizia (*Paraserianthes falcataria* (L.) Nielsen) and gmelina (*Gmelina arborea* Roxb.) woods planted in Sabah, Malaysia. For each thickness of the blocks, the radial surfaces were glued and edgejointed together with resorcinol resin (Aica Co.) and pressed at room temperature for 24h to produce 300 (L) × 300 (T) mm blocks with thicknesses of 10, 15, and 20 (R) mm. To compare them to Japanese wood, edge-jointed hinoki (*Chamaecyparis obtusa* Endl.) lumber 300 × 300 mm with thicknesses of 10, 15, and 20 mm obtained from Megumi Panel Co. were used. The average moisture content of all the lumber was 9%. The densities of the albizia, gmelina, and hinoki lumber were 0.30, 0.50, and 0.51 g/cm³, respectively. For each condition, two specimens (one untreated and one treated) were prepared for the JIS A 1304 test.

Another set of edge-jointed lumber from albizia and gmelina woods (planted in West Java, Indonesia) and hinoki wood was prepared for the cone calorimeter test. The glue and the procedure to make the lumber were the same as described above. The lumber was prepared at 600 (L) \times 300 (T) mm with thicknesses of 10, 15, and 20 (R) mm and was then cut into 100 (L) \times 100 (T) mm specimens to meet the required sample size for the cone calorimeter test (ISO 5660). The densities of albizia, gmelina, and hinoki lumber were 0.27, 0.44, and 0.49 g/cm³, respectively. The average moisture content of the lumber was about 8%–10%. For each condition, four specimens (two untreated, two treated) were prepared.

The fire-retardant chemical was prepared by mixing trimethylol melamine powder and an 85% solution of phosphoric acid and water. The molar ratio was 1.0:1.5, and water was added to make a 25% aqueous solution. The resulting solution was clear and had a pH of 2.5. All treated lumber was coated on one surface (tangential surface) with fire-retardant chemical in the amount of 100 g/m². This amount alone was used because the purpose was to compare untreated and fire-retardant-treated specimens. Some studies showed that a coating of trimethylol melamine phosphoric acid at 100 g/m² significantly improved the fire retardancy of wood compared to untreated lumber.^{4,10} The effect of the amount of coating using trimethylol melamine phosphoric acid on the fire retardancy of wood had been studied previously. 10 The chemicals were simply spread over the surface of the specimens to coat them, and then the specimens were cured at room temperature for 4 weeks. Heating or curing was not attempted in the experiment, and pressure was not applied. This coating method has also been used in other studies.3,4

Fire test methods

Two fire test methods were performed. For the first, the lumber was tested in a laboratory-scale exposure furnace in accordance with JIS A1304. The laboratory-scale furnace was a modification of a full-scale furnace designated to test the structural parts of buildings with a minimum sample size of $900 \, \text{mm}$. Lumber of $300 \times 300 \, \text{mm}$ was held in a steel

holder and set in the vertical position. The lumber was exposed to fire from propane gas. The heating temperature inside the furnace was controlled by adjusting the amount of gas and air to follow the time-temperature curve as required by the standard. The exposed and unexposed temperatures were measured using thermocouples. They were measured at one point on the center of the exposed and unexposed surfaces of the specimen. The size of the specimen tested in this experiment (i.e., $300 \times 300 \,\mathrm{mm}$) was smaller than the minimum size of specimens (900 \times 900 mm) cited by JIS A 1304 (size of test piece type C), which required a minimum of three measurement points. The time it took to reach a temperature of 260°C at the unexposed surface of sample and to burn through were recorded.

For the second test, the lumber was tested with the cone calorimeter followed the ISO 5660 standard. The weight and thickness of the 100×100 mm lumber were determined before testing. The lumber was wrapped in an aluminum foil sheet and left open on an exposed surface. The lumber was exposed to a constant heat flux of $40 \,\mathrm{kW/m^2}$ on one surface. A spark igniter was applied to ignite the lumber. The tests were conducted at a horizontal orientation, with the sample set in a retainer frame. The parameters observed were time to ignition and mass loss rate. The cone calorimeter at our laboratory in Indonesia does not accommodate a heat release rate measurement; instead, we used thermocouples to measure the rise of temperature on the sample. Thermocouples were placed on the exposed and unexposed surfaces of the sample and connected to a data logger to record the temperature. The exposed and unexposed temperatures were measured at one point on the center of the exposed and unexposed surfaces of the specimen.

Results and discussion

Temperatures of exposed and unexposed lumber in the fire tests

Temperatures on the exposed lumber surfaces during the JIS A 1304 and cone calorimeter tests are compared in Fig. 1. The specimens measured were treated hinoki lumber with a thickness of 20mm for both the laboratory furnace JIS A 1304 and the cone calorimeter tests. The temperatures of other exposed specimens from different wood species, different thicknesses, and treated or untreated behaved similarly. The reason might be, for JIS A 1304, that the source of heat was adjusted to produce heating temperatures inside the furnace that follow the time-temperature curve as required by the standard and for the cone calorimeter test that the heat flux was set constant at 40 kW/ m². The temperature of the exposed lumber from the cone calorimeter test rose quickly to about 800°C just after the beginning of the test owing to ignition on the lumber surface. It maintained temperatures of 800°-850°C during the test because of the constant heat flux of $40 \,\mathrm{kW/m^2}$ to which the lumber was exposed. After ignition the lumber burned

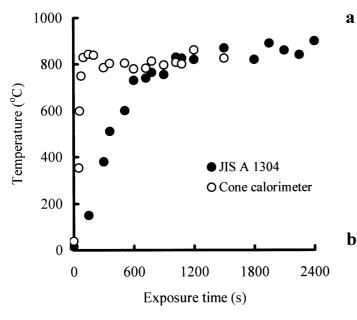
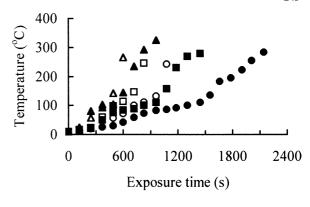


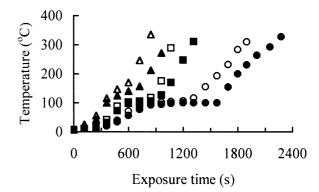
Fig. 1. Temperature of the exposed surfaces of treated hinoki lumber with a thickness of 20 mm in the JIS A 1304 and cone calorimeter tests

at high flame, although gradually the flame diminished and remained at a constant, low flame for some time. The flame then gradually increased again to become a high flame, followed by a gradual decrease until it finally burned out. The exposed temperature from the JIS A 1304 test gradually increased to 730°C at 10 min, 820°C at 20 min, and about 850°-900°C after 20 min until the end of the test. Except for the difference at the beginning of the test, the temperatures at the exposed surface of sample are reasonably comparable between the two tests, especially up to 30min of the test. Tsantaridis and Ostman, 16 though, suggested that a higher heat flux of 50 kW/m² is roughly more equivalent to the ISO 834 standard time-temperature curve during the first 30-40 min of the test. The JIS A 1304 is analogous to the ISO 834. However, it should be noted here that during the cone calorimeter test the heat was applied as irradiance heat (without a flame) in contrast to the JIS A 1304, where the heat was applied in the form of flames. The difference in the type of heat applied might produce different effects on the fire properties of the lumber.

Figure 2 shows the temperatures of unexposed surfaces from untreated and treated albizia, gmelina, and hinoki lumber of different thicknesses during the JIS A 1304 test. For all the lumber the temperature rose slowly to 100°C and leveled off at this temperature for some time. This time delay in temperature at about 100°C might be due to the evaporation of moisture in the lumber. The leveling off of the temperature curve was longer for the treated lumber than for the untreated lumber. The treated lumber exhibited less temperature rise, and increasing the lumber thickness decreased the temperature rise.

The temperatures of unexposed surface untreated and treated albizia, gmelina, and hinoki lumber at various thicknesses during the cone calorimeter test are shown in Fig. 3. Treatment with a fire retardant delayed the temperature





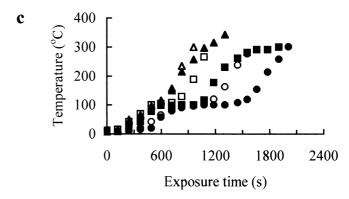
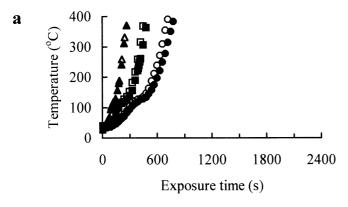
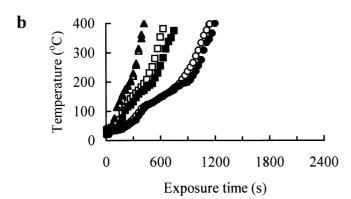


Fig. 2. Temperatures of unexposed surfaces of untreated (*open symbols*) and treated (*filled symbols*) albizia (**a**), gmelina (**b**), and hinoki (**c**) lumber in the JIS A 1304 test. Lumber thickness: *triangles*, 10 mm; *squares*, 15 mm; *circles*, 20 mm

rise at the surfaces of the unexposed lumber in all wood species at all thicknesses. Temperatures rose faster in albizia lumber than in gmelina or hinoki lumber; density may play a role here. Lumber thickness also altered the temperature rise, with thicker lumber exhibiting less temperature rise.

The temperatures of unexposed surfaces rose faster in the cone calorimeter test (Fig. 3) than in the JIS A 1304 test (Fig. 2). In the cone calorimeter test a high flame developed after ignition and the lumber surface burned quickly; at the same time, exposure to radiant heat from the cone heater was continuous. The quick temperature rise at the





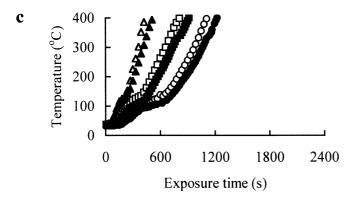


Fig. 3. Temperatures of unexposed surfaces of untreated and treated albizia (**a**), gmelina (**b**), and hinoki (**c**) lumber at various thicknesses in the cone calorimeter test. See Fig. 2 for symbols

beginning of the test is expressed as the "exposed temperature" (Fig. 1). In the JIS test the temperature in the furnace rose gradually (Fig. 1), which caused a slower "unexposed temperature" rise than in the cone calorimeter test. In the cone calorimeter test the temperature leveled off at 100°C but to a lesser extent than in the JIS test.

Effects of wood species, lumber thickness, and treatment on fire-retardant properties

To compare the effects of wood species, lumber thickness, and treatment on fire-retardant properties, the surface den-

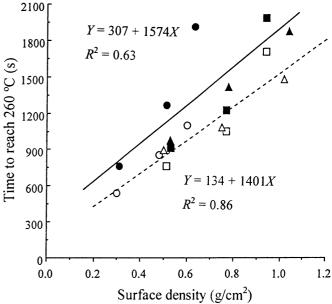


Fig. 4. Effects of surface density on the time to reach 260°C on the unexposed surface of the sample in the JIS A 1304 test. *Circles*, albizia; *squares*, gmelina; *triangles*, hinoki; *filled symbols*, treated lumber; *open symbols*, untreated lumber

sity (i.e., mass per unit area, or a product of density and thickness) was introduced. Figure 4 shows the effects of surface densities on the times required to reach a temperature of 260°C at the unexposed surface of albizia, gmelina, and hinoki lumber during the JIS A 1304 test. A temperature of 260°C is considered the critical temperature at which cellulose starts to degrade. The time to reach 260°C at the unexposed surface of lumber increases with increasing surface density of the lumber. The same result has been found for wood-based panels (particleboard, plywood) tested at small and full scale in accordance with JIS A 1304. 14,15 In a small-scale test, particleboard (density 0.7 g/cm³, thickness 6–30 mm) with a surface density of 1 g/cm² is required for it to take about 30 min to reach 260°C on the unexposed surface of the lumber. 15 That result is comparable to the results obtained in this study (Fig. 4). The treated lumber takes about 20% longer than untreated lumber to reach 260°C on the unexposed surface. Formation of a char layer on the lumber surface due to the reaction of the chemicals prevents the increase in temperature.

Figure 5 shows the effects of surface density on burnthrough times in the JIS A 1304 test. Similar to the results of time to reach 260° C on the unexposed surface of lumber, the burn-through time of treated lumber was 30%-40% longer than that of untreated lumber. This result on edge-jointed lumber is in agreement with results from sugi laminated boards. ¹⁰

The effects of surface density on ignition times of the lumber in the cone calorimeter test are shown in Fig. 6. In general, untreated lumber ignites faster than treated lumber. Similar results using the same compounds have been observed by others. Ellis and Rowell stated that chemicals containing phosphorous compounds reduced the

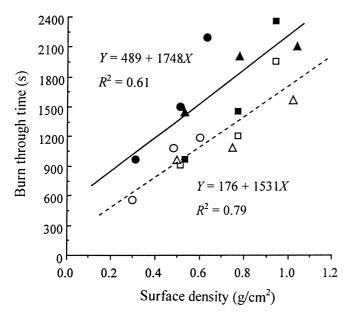


Fig. 5. Effects of surface density on burn-through times in the JIS A 1304 test. See Fig. 4 for symbols

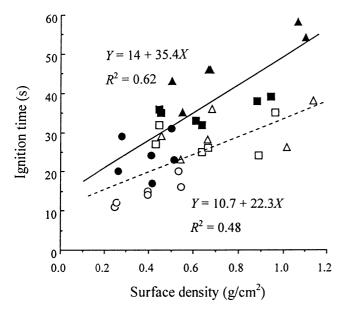


Fig. 6. Effects of surface density on ignition times in the cone calorimeter test. See Fig. 4 for symbols

pyrolysis temperature, which in turn suppressed the formation of volatile combustible materials. In this experiment, the time of ignition of treated lumber was delayed owing to the formation of char on the surface layer. Figure 6 shows the tendency that increasing surface density increases the ignition time. Harada¹⁹ found in some untreated Japanese woods that ignition time is strongly correlated with density, increasing linearly with increasing density. Albizia exhibited a short ignition time, whereas gmelina and hinoki behave similarly; it is related to the effect of lumber density.

Figure 7 shows effects of surface density on the time required to reach 260°C on the unexposed surface of albizia, gmelina, and hinoki lumber in the cone calorimeter

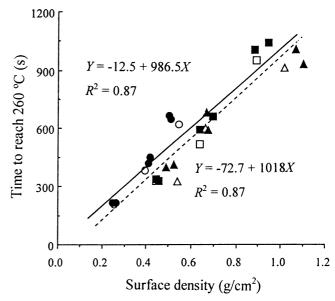
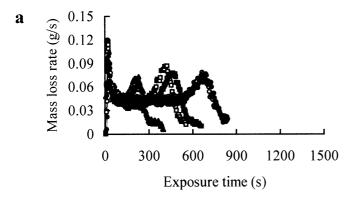
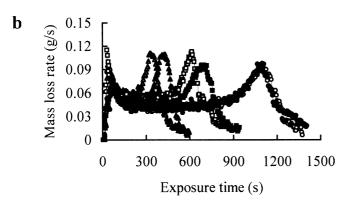


Fig. 7. Effects of surface densities on the time required to reach 260°C on an unexposed surface of the sample in the cone calorimeter test. See Fig. 4 for symbols

test. It was found that an increase in surface density increased the time to reach 260°C on the unexposed surface of lumber. Treated and untreated lumber data showed no difference in the time needed to reach 260°C on the unexposed surface of lumber; generally, however, treated lumber took a longer time than untreated lumber. A similar trend was seen with the results of the JIS A 1304 test (Fig. 4), although the time needed to reach 260°C on the unexposed surface of lumber was less in the cone calorimeter test. The results of the JIS A 1304 test were almost double those of the cone calorimeter test. For example, the shortest times required to reach 260°C on the unexposed surface of lumber were 206 and 541s for untreated albizia lumber and the longest times were 1039 and 1981s for treated gmelina lumber in the cone calorimeter and the JIS A 1304 tests, respectively.

Figure 8 shows mass loss rates of untreated and treated albizia, gmelina, and hinoki lumber at various thicknesses in the cone calorimeter test. The mass loss rate (MLR) is one of the indicators of fire endurance of lumber: the higher the MLR, the more material is burned and the less endurance there is to fire. As commonly observed in wood or woodbased products, the MLR curve during the test can be divided into three stages.¹⁹ At the beginning of the test, condensed smoke is released from the lumber before ignition occurs. High flame occurs just after ignition, and the MLR increases to reach its highest peak, called the first MLR peak (first stage). The MLR then gradually decreases as the flame decreases and char develops. The MLR remains the same for some time as the flame becomes constantly smaller (second stage). The flame then gradually increases again, as does the MLR, until the MLR reaches a second peak (third stage). The flame then gradually decreases again (as does the MLR), and the flame is extinguished when the lumber burns out.





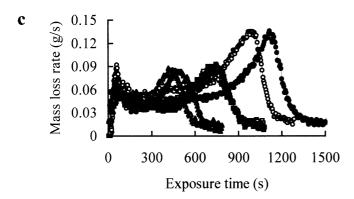


Fig. 8. Mass loss rates (MLRs) of albizia (a), gmelina (b), and hinoki (c) lumber in the cone calorimeter test. See Fig. 2 for symbols

In general, the MLR of treated lumber was lower than that of untreated lumber; the treatment prolongs the time needed to reach the second MLR peak. The effects of thickness can be seen by examining the different times needed to reach the second MLR peak (i.e., thicker lumber needs more time to reach the peak). At the same thicknesses, albizia lumber burned out faster than gmelina or hinoki lumber. Further analyses (see below) were conducted for the MLR of the first peak and the time required to reach the second MLR peak.

Because the early fire properties are important from a fire safety point of view, the first MLR peak was analyzed further. Figure 9 shows the effect of surface density on the first MLR peak. The MLR of untreated lumber was higher than that of treated lumber, so it seems that treatment

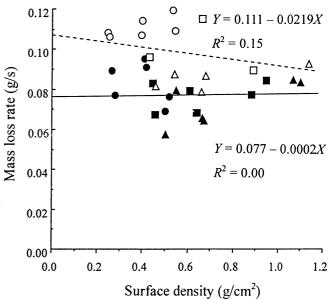


Fig. 9. Effects of surface density on the first MLR peak in the cone calorimeter test. See Fig. 4 for symbols

suppressed the fire.¹⁷ There was no correlation between surface density and MLR in the treated lumber, and a similar trend has been observed in solid wood.¹⁹ The chemical treatment resulted in homogeneous fire behavior of the lumber during the initial stage. The untreated albizia lumber exhibited the highest MLR, followed by gmelina and hinoki lumber, whereas the treated lumber of albizia, gmelina, and hinoki exhibited almost the same MLR range (0.06–0.10 g/s). The difference in surface densities therefore did not affect the first MLR peak of the treated lumber.

Together with the critical temperature of 260°C (when cellulose starts to degrade), a temperature of 300°C attracts special interest because it is the temperature at which wood starts to char. 16,20,21 In this experiment the temperature data were obtained from the cone calorimeter test by attaching thermocouples to the exposed and unexposed surfaces of the lumber. An attempt was made to relate the time needed to reach 300°C on the unexposed surface and the time to reach the second MLR peak. Here we looked at the start of carbonization of the unexposed surface of lumber, which is assumed to occur when the temperature of the lumber reaches 300°C. The results for the untreated and treated lumber are presented in Fig. 10. It was found that the time to reach the second MLR peak is strongly correlated with the time to reach 300°C on the unexposed surface of the sample. The correlation can be expressed as follows:

$$T = 0.29 + 1.02TS (R^2 = 0.99)$$

where T is the time to reach a temperature of 300° C on the unexposed surface of the sample (the time at which wood starts charring), in seconds; TS is the time to reach the second MLR peak, in seconds; and R^2 is the coefficient of determination. It was confirmed from this experiment that the time to reach the second MLR peak can be assumed to be the time when the wood or wood-based product starts to char.

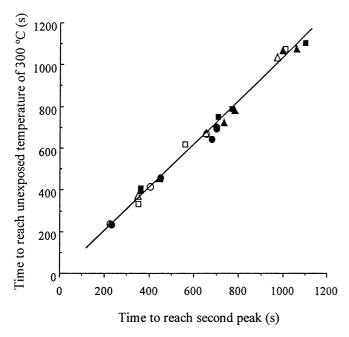


Fig. 10. Relation between the time required to reach the second MLR peak and the time required to reach 300°C on the unexposed surface in the cone calorimeter test. See Fig. 4 for symbols

Conclusions

The fire endurance of edge-jointed lumber from tropical fast-growing albizia and gmelina and hinoki woods coated with a solution of phosphoric acid melamine formaldehyde compound was discussed. Cone calorimetry and a standard fire tests were used to evaluate the performance of the lumber. The results showed that the fire endurance of the lumber was improved by the treatment. The results of the cone calorimeter and standard fire tests were comparable, although the cone calorimeter test was slightly more rigorous for the lumber.

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